Uniaxial Strength and Mode I Fracture Toughness of Hot Pressed Silicon Carbide (SiC) Materials

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Abstract

Silicon carbide (SiC) ceramics are being used and considered for a number of room temperature wear and optical applications where strength and fracture toughness are critical design parameters. Thus, there is a requirement for high-quality tensile strength and Weibull data for this class of materials. This report will present room temperature uniaxial tensile strength data, single-edge precracked beam (SEPB) fracture toughness (K_{IC}) data, and fractographic analysis for several hot-pressed (HP) SiC ceramics. Strength and K_{IC} data will be related to the micro structure and flaw population of the HP SiC materials studied.

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1. Introduction

The high elastic modulus, high hardness, excellent wear and corrosion resistance, low density, and good specific strength of silicon carbide (SiC) have led to its use in room temperature wear parts, optical components, and armor applications. The increasing use of SiC ceramics in such applications demands reliable strength statistics and accurate fracture toughness data as critical design parameters. Such information also provides valuable feedback to materials processing, which can lead to increased product quality and reliability. While much data exist on the strength and toughness of sintered SiCs [1–3] and some data are available on an older hot-pressed (HP) version of SiC [4], little data exist on newer HP varieties.

This work presents a detailed investigation of the room temperature tensile and flexural strength, failure statistics, and room temperature fracture toughness of two different types of HP SiC materials. Observations on the strength limiting flaws are based on fractography of tensile specimens. The possible effects of varying machining practices on the strength and Weibull statistics data were examined by having sets of specimens machined by two different vendors. Weibull statistics obtained from the tensile specimens were employed to predict strength values obtained from four-point bend tests. One of the two materials was provided as both small and very large billets, thus the effects of billet size could be studied. Increasing billet size has been demonstrated to affect the strength of Si_3N_4 [5], thus it is important to ascertain whether such a phenomenon is present in HP SiC.

2. Materials

The SiC materials used in this study were Cercom* PAD SiC-B and PAD SiC-N, which will be hereafter designated as SiC/B and SiC/N, respectively. These SiCs were hot-pressed into 15.24- × 15.24- × 2.54-cm billets. One additional Cercom PAD SiC-B material was fabricated into a 31.75- ×

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* Cercom, Inc., Vista, CA.

31.75- × 17.27-cm large billet and will be designated as SiC/B LB. Both PAD SiC-B and PAD SiC-N materials are reported [6] to have almost identical mechanical and physical properties except that PAD SiC-N has improved impact and dynamic properties. The manufacturer reported a bulk density of 3.2 g/cm³, average grain size of 4.0 µm, four-point flexure strength of 655 MPa, Weibull modulus of 18, elastic modulus of 455 GPa, Poisson's ratio of 0.14, and fracture toughness of 5.2 MPa√m [7]. X-ray diffraction patterns obtained from powdered samples indicated that the major phase was hexagonal α SiC. A trace of a rhomobohedral α SiC was present in the SiC/B, whereas no such phase was present in the SiC/N. Direct detection of a cubic β SiC phase was unsuccessful because of overlapping peaks between the α and β phases. However, comparison of the measured integrated intensities and those listed in the JCPDS cards indicated no JCPDS presence of β SiC phase in either material.

3. Experimental Procedure

Two different highly reputable machining vendors were used to fabricate SiC/B and SiC/N modulus of rupture (MOR) and tensile specimens. Vendors A and B prepared 30 B-type MOR specimens from each material according to MIL-STD-1942a [8]. Vendor A prepared 16 tensile specimens from both SiC/B and SiC/N, whereas Vendor B prepared 15 and 14 tensile specimens from SiC/B and SiC/N, respectively. The tensile specimens were fabricated by grinding the specimens circumferentially while meeting the same surface finishing procedures and requirements as the MOR specimens. Nominal dimensions of the tensile specimens were 8.8 mm in diameter and 120 mm in length. Both the MOR and tensile specimens from SiC/B LB were fabricated from the inside portion of the billet by Vendor A.

Tensile tests were performed at room temperature using a self-aligning hydraulic testing apparatus developed by Baratta and Driscoll [9] with a simplified specimen geometry described by Hermansson et al. [10], which is a simple right cylinder, nominally 9 mm in diameter and 120 mm long. On each end of the tensile specimens, 40 mm are inserted into steel pistons and adhesively

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bonded in place with a high-strength epoxy.[*] The specimen-piston assembly is inserted into the pressure chamber of the hydraulic tester.[**] Pressure is applied and increased until the specimen in broken apart by the hydraulic pressure acting against the pistons. A detailed description of this test method may be found in the Katz, Lucas, and Toutanji [3], Hermansson, Adlerborn, and Burstrom [10], Lucas [11], Toutanji [12], and Katz et al. [13].

Lucas [11] and Toutanji [12] describe a technique for correcting small amounts of eccentricity in loading. All data presented in this report were subjected to such correction procedures. Typically, in this study, such corrections represent only 2% of the nominal stress (maximum of 5%). Due to the effects of a stress concentration at the specimen-to-piston bond transition, data from specimens that fractured within one-half the radius of the specimen from the epoxy line were not considered valid tests. Such data, however, can be used as censored (or suspended) data points from the Weibull statistics calculation [14], as will be described in the following paragraphs.

The fracture surface of tensile specimens was examined by low-magnification optical and high-magnification scanning electron microscopy (SEM). SEM fractography and elemental analysis were carried out using a JEOL 840A instrument equipped with a KEVEX energy dispersive x-ray analyzer. The tensile fracture surfaces were ultrasonically cleaned and coated with a thin layer of carbon or Au-Pd alloy to prevent charging.

Four-point bend tests were carried out in accordance with MIL-STD-1942a [8] using a fully articulating fixture having 20-mm inner and 40-mm outer spans. An Instron screw-driven 25-kN load-capacity universal test machine with a 5-kN capacity load cell was used. All specimens were fractured at a displacement rate of 0.5 mm per minute. Fractographic analysis of the MOR specimens will not be reported in this study.

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[*] ARALDITE AV 118, Ciba Geigy Corp., East Lansing, MI.
[**] ASCERA Hydraulic Tensile Tester, Robertsfors, Sweeden.

Weibull statistical data for MOR and tensile tests were obtained from the SiC/B and SiC/N specimens machined by Vendors A and B. One additional data set for each test method was obtained from the SiC/B LB specimens. Weibull modulus, m, and Weibull characteristic strength, σ_{CH}, were estimated using the two-parameter Weibull equation [15] by fitting the data using the maximim likelihood method [16]. Additionally, the effects of performing a censored data analysis on the tensile Weibull data was evaluated following the procedure described in Abernethy et al. [14]. In a censored data Weibull plot, the existence of the invalid tests is taken into account in ranking the data. This, therefore, affects the values for the probability of failure for each valid test.

Fracture toughness, K_{IC}, estimation was performed only on the tensile specimens having internal failure initiating flaws with fully developed fracture mirrors [17]. Flaw size was measured from high magnification SEM photomicrographs. Fracture toughness was estimated following the Sneddon solution [18]:

$$K_{IC} = 2\,\sigma_c\,(a_c/\pi)^{1/2}, \tag{1}$$

where K_{IC} is the fracture toughness, σ_c is the fracture stress, and a_c is one-half the critical flaw size.

Broken halves from the four-point MOR tests were used for SEPB K_{IC} tests using the experimental technique described in Nose and Fujii [19], Bar-On [20], and Quinn [21]. The specimens were fractured in three-point bending at a displacement rate of 0.5 mm per minute using an Instron 5-kN load capacity universal testing machine with a 0.5-kN load cell. Fracture toughness was calculated from Srawley's stress intensity solution [22] using a span-to-width ratio of 4 and following the precrack length measurement procedure described in ASTM STD E399 [23].

4. Results and Discussion

4.1 Tensile Strength Distribution. Table 1 summarizes the Weibull statistics and failure initiation sites for the tensile tests. The data show that a significant (~11%) variation in tensile

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Table 1. Tensile Weibull Statistics and Failure Initiating Flaw Population Data

Material	Not Censored			Censored			Failure Origin
	σ_{CH}(MPa)	m	No./Sus.	σ_{CH}(MPa)	m	No./Sus.	vf/ss/s
SiC/B LB	304	9.6	16/0	306	9.8	18/2	10/3/5
SiC/B VA	399	16.6	14/0	399	16.7	16/2	1/0/15
SiC/B VB	357	19.0	13/0	357	19.1	15/2	0/3/12
SiC/N VA	359	10.9	14/0	367	10.1	16/2	4/2/10
SiC/N VB	318	15.7	8/0	325	16.3	14/6	2/4/8

Notes:
vf = volume flaws.   ss = subsurface flaws.
s = surface flaws.   No. = total number of specimens.
Sus. = number of suspended items.   LB = larger billet.
VA = vendor A.   VB = vendor B.

strength can result from machining variation. More than 50% the SiC/B LB specimens failed from internal volume flaws. SiC/B specimens, on the other hand, predominantly failed from either surface or subsurface flaws regardless of the machining vendor. The greater prevalence of internal failure origins combined with the much lower characteristic strength and Weibull modulus (m) of the large billet material is indicative or a significant increase in volumetric, process-related flaws, as compared to the smaller billets. Of the specimens from the smaller billets, only 1 of 31 valid failures of SiC/B occurred from an internal flaw, whereas SiC/N material had 6 internal flaws for 30 valid tests. The greater frequency of internal flaws coupled with the slightly lower value of σ_{CH} and m for the SiC/N material may be explained by the fact that this material is a more recent development than SiC/B and, thus, may be less "process mature." Nevertheless, the tensile strength observed in both of these HP SiCs is impressive as compared to previously reported tensile strengths of SiC. Miller et al. [4] reported an average room temperature tensile strength of 270 MPa for an early 1970s HP SiC (NC-203).* Reported characteristic tensile strength of sintered SiCs ranges from 213 MPa with an "m" of 7.5 [24] to 307 MPa with an "m" of 9.4 [3].

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* Norton Co., Worcester, MA.

The censored data concept [14] was used to readjust ranking of data points by accounting the specimens which failed within one-half of the radius from the glue line. The results are summarized in Table 1. Censored Weibull statistics were essentially the same as those obtained from the uncensored data. Since the differences between using uncensored-vs.-censored data analyses were minimal, uncensored values were used in making Weibull predictions, in keeping with past practice [13]. Figure 1a presents the uncensored tensile Weibull distributions of the SiC materials used in this study.

4.2 Modulus of Rupture (MOR) Strength Distribution. Results of the MOR Weibull strength statistics are presented in Table 2. Higher values of characteristic strength and Weibull moduli were observed compared with those obtained from tensile tests. This observation is expected based on the well-known stressed volume effects on Weibull parameters. Increased values of Weibull modulus, m, were also observed for the MOR specimens. The MOR strength varied less as a function of machining vendor than did the tensile strengths. This observation may be related to the fact that the machining parameters for fabrication of MOR specimens are well-established, whereas similar parameters are needed to be established for the fabrication of tensile specimens. Figure 1b presents the Weibull plots for the MOR specimens evaluated in this study.

4.3 SEPB K_{IC} Test. Fracture toughness data for the SiC materials used in this study are listed in Table 3 along with the values taken from the literature [17, 25–27]. Convincing evidence of significantly improved fracture toughness of newer grades of HP SiC, as compared to earlier HP or sintered materials, is evident. Hot-pressing of SiCs into very large billets, however, tends to decrease not only the strength but also the fracture toughness as evidenced by K_{IC} values measured from SiC/B LB and SiC/B materials. A marginal improvement in fracture toughness was exhibited by the SiC/B compared with the value obtained for the SiC/N. Again, the higher standard deviation in measured toughness value of the SiC/N material supports the earlier stage of material maturity described in the previous section. The "new" version of the SiC/B material evaluated in this and previous work [25] shows a marked improvement in K_{IC} over the "older" version of the same
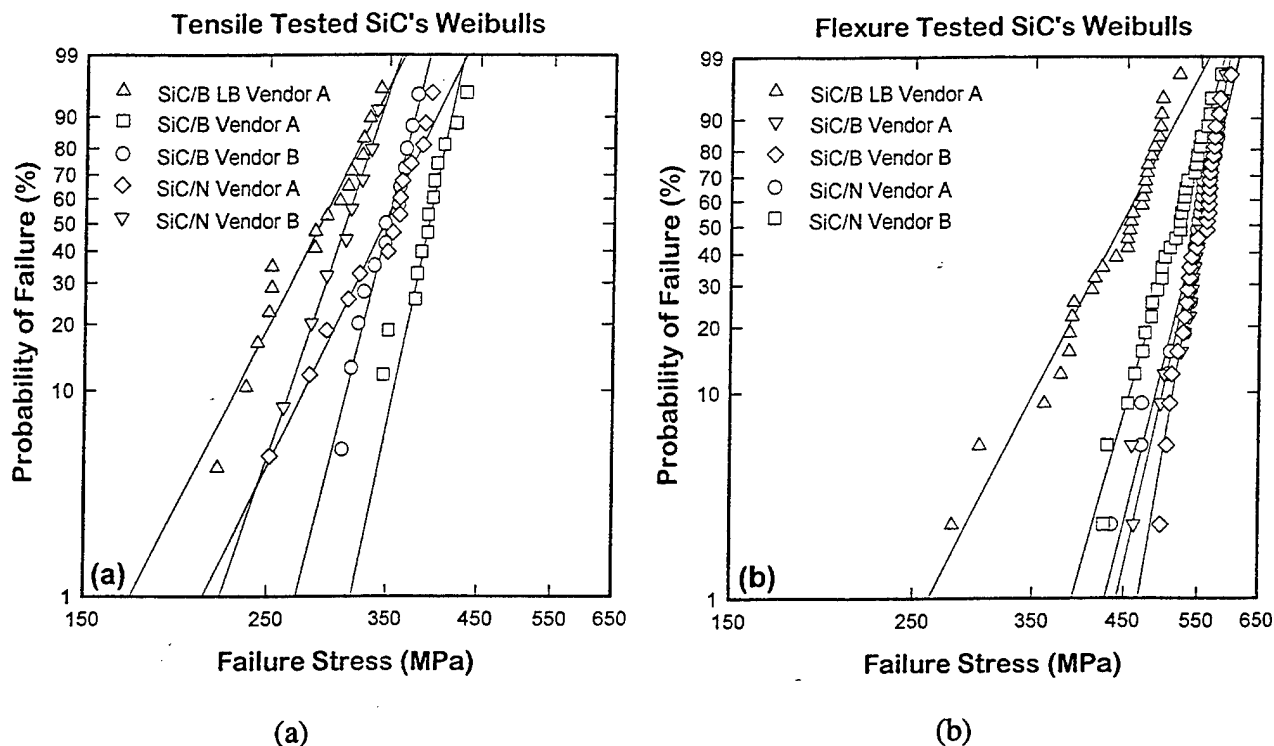
Figure 1. Weibull Plots of the Uncensored (a) Tensile and (b) Four-Point Flexure Tested Specimens for the Five Test Conditions Studied.

material [26]. The fracture toughness of SiC/B and SiC/N is now of the same order as NC-132[*] Si_3N_4 [28].

4.4 Estimation of K_{IC} From Fractography. The tensile fracture surfaces of the SiC/B LB material yielded 10 samples that failed from volume defects, had well-developed fracture mirrors, and met the other cirteria specified in Katz et al. [17]. Similarly, the SiC/N material produced five samples meeting these conditions. The SiC/B samples only yielded one such fracture surface, which was not analyzed, as no statistics could be developed from a single estimation. Figure 2 shows a

[*] Norton Co., Worcester, MA.

typical failure origin in the SiC/B LB material. The estimated values of K_{IC} were 4.1 ± 0.8 MPa√m and 4.3 ± 0.4 Mpa√m for the SiC/B LB and SiC/N materials, respectively. These estimated values differ by 2.5% and 12% from the SEPB measurements. Such variances are comparable to those observed for other materials evaluated in this manner [17].

Table 2. Weibull Strength Data From the Four-Point MOR Specimens

Material	σ_{CH}(MPa)	m	No. of Specimens
SiC/B LB	460	10.5	30
SiC/B VA	560	27.3	30
SiC/B VB	560	26.7	30
SiC/N VA	556	22.8	30
SiC/N VB	529	26.7	30

Table 3. K_{IC} Comparison of SiC Materials

Material	Manufacturer	Method	Mean K_{IC}	Reference
HP SiC/B LB	Cercom	SEPB	4.00 ± 0.27	This Study
HP SiC/B[a]	Cercom	SEPB	5.09 ± 0.28	This Study
HP SiC/N	Cercom	SEPB	4.92 ± 0.69	This Study
HP SiC/B[a]	Cercom	SEPB	4.18 ± 0.61	Cho et al. [25]
HP SiC/B[a]	Cercom	Chevron Notch	4.57 ±0.49	Cho et al. [25]
HP SiC/B[a]	Cercom	Indentation Strength	4.01 ± 0.29	Cho et al. [25]
HP SiC/B[b]	Cercom	SEPB	2.20 ± 0.60	Mariano et al. [26]
Sintered SiC	Dow	SEPB	3.11 ± 0.33	Katz et al. [17]
Sintered SiC	Ford	SEPB	2.30 ± 0.25	Woodilla et al. [27]

[a] New
[b] Old
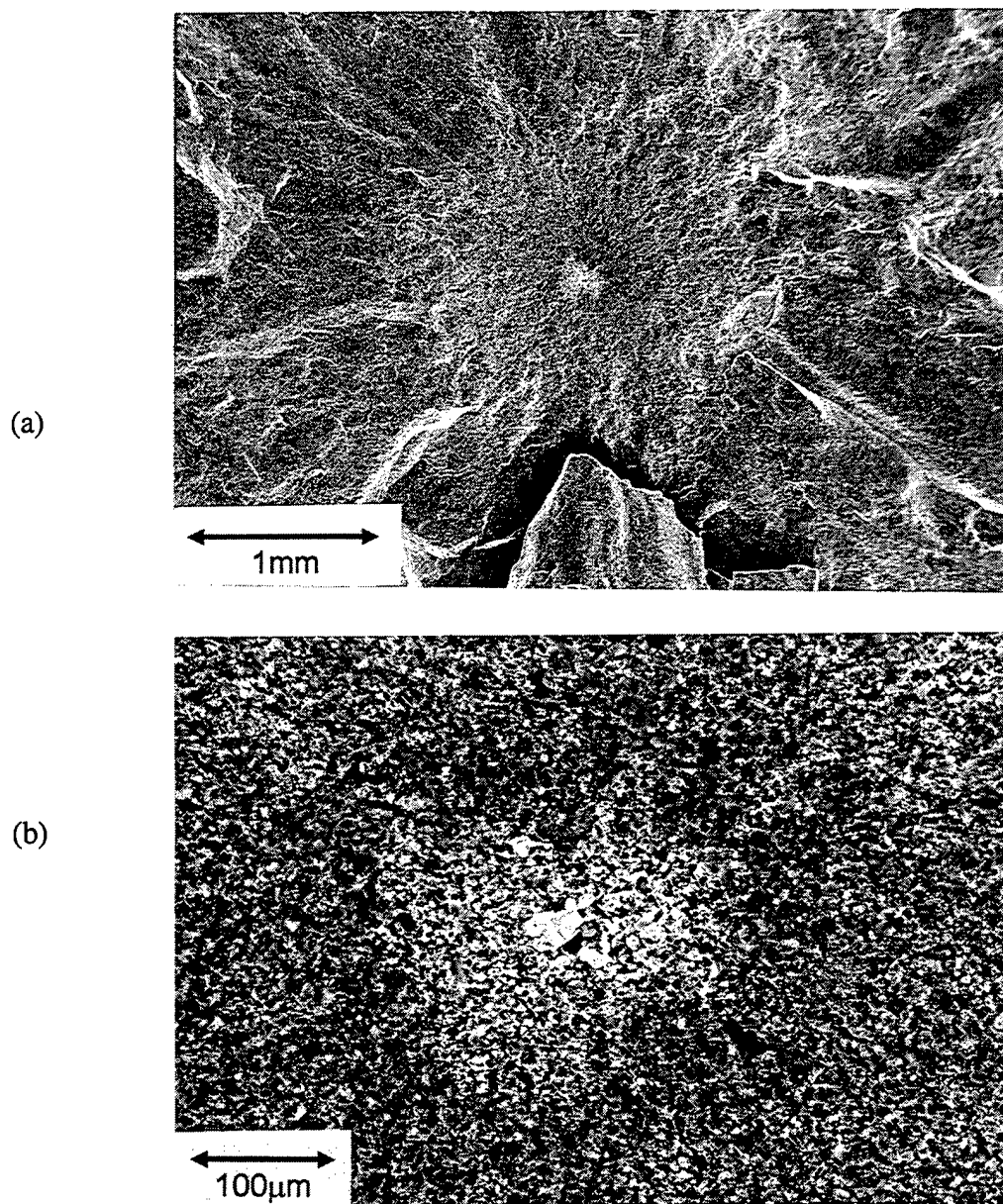
Figure 2. SEM Fractographs of Specimen of SiC/B LB (a) Volume Flaw, Fracture Surface Shown Failure Origin With Distinct Circular Mirror, Mist, and Hackle Regions; (b) Failure Origin Consists of a Cluster of Large Grains.

4.5 Prediction of MOR Weibull Strength From Tensile Weibull Parameters. Theoretically, Weibull characteristic MOR strength values can be predicted from tensile Weibull parameters by equating the unit volume characteristic strength [29] of tensile and flexure specimens. Table 4 presents predicted and measured strength values in this study. The instance of 13% disagreement between predicted and measured MOR values may indicate that fracture occurs from different flaws

(most likely, surface flaws of different orientations resulting from axial vs. circumferential) than in the case of the tensile bars. In all instances, the measured MOR values are higher than the ~400 MPa previously reported by Mariano and Bar-On [26].

Table 4. Predicted and Measured Weibull Characteristic MOR Strength

Material	Tensile		MOR		Difference (%)
	σ_{CH}	m	Predicted	Measured	
SiC/B LB	304	9.6	524	460	-14
SiC/B VA	399	16.6	565	560	-1
SiC/B VB	357	19.0	487	560	13
SiC/N VA	359	10.9	587	556	-6
SiC/N VB	318	15.7	458	529	13

5. Conclusions

The conclusions of this study are as follows:

(1) Tensile strength of both HP SiC/B and SiC/N is significantly higher than previously reported variants of SiC. Similarly, the fracture toughness of these grades of SiC is also higher and is comparable to NC-132 grade of HP Si_3N_4.

(2) Scaling up the volume of HP SiC billets by ~30 × was observed to decrease average tensile strength by ~15–24%, and MOR by ~18%.

(3) Machine-shop-to-machine-shop practice variation can lead to an ~11% difference in measured tensile strengths, as opposed to only a 0–5% variation in MOR values.

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(4) Using a censored-vs.-uncensored Weibull analysis made no significant difference for any of the five conditions evaluated.

(5) Estimation of fracture toughness by fractographic analysis yields values within 12% of SEPB fracture toughness measurements.

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Uniaxial Strength and Mode I Fracture Toughness of Hot-Pressed Silicon Carbide (SIC) Materials

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Silicon carbide (SiC) ceramics are being used and considered for a number of room temperature wear and optical applications where strength and fracture toughness are critical design parameters. Thus, there is a requirement for high-quality tensile strength and Weibull data for this class of materials. This report will present room temperature uniaxial tensile strength data, single-edge precracked beam (SEPB) fracture toughness (K_{IC}) data, and fractographic analysis for several hot-pressed (HP) SiC ceramics. Strength and K_{IC} data will be related to the micro structure and flaw population of the HP SiC materials studied.

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